

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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TECHNOLOGIC PAPERS OF THE BUREAU OF STANDARDS, No. 364

[Part of Vol. 22]

**TENSILE PROPERTIES OF SOFT RUBBER COMPOUNDS  
AT TEMPERATURES RANGING FROM  
-70° TO +147° C.**

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*Bureau of Standards*

January 21, 1928



PRICE 10 CENTS

\$1.25 PER VOLUME ON SUBSCRIPTION

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UNITED STATES  
GOVERNMENT PRINTING OFFICE  
WASHINGTON

1928



# TENSILE PROPERTIES OF SOFT RUBBER COMPOUNDS AT TEMPERATURES RANGING FROM $-70^{\circ}$ TO $+147^{\circ}$ C.

By R. F. Tener, S. S. Kingsbury, and W. L. Holt

## ABSTRACT

This paper gives the results of tensile tests of six rubber compounds when tested at temperatures ranging from  $-70^{\circ}$  to  $+147^{\circ}$  C. Photographs are included showing the apparatus employed and the methods used are described. The results are shown on 13 graphs. The first 6 show tensile strengths and elongations at break for each compound, the second 6 stress-strain data, and the last 1 tensile products. The results show that temperature has a very pronounced influence on the tensile properties of rubber. At low temperatures rubber becomes rigid with an increased tensile strength and at high temperature it possesses practically no tensile strength. The various rubber compounds investigated show the same type of behavior with changes in temperature. Differences due to compounding ingredients are differences in degree rather than in kind. The magnitude of the changes in rubber compounds due to temperature shows that the effect of temperature is a feature which should be given consideration in the design of rubber articles which are used at other than normal temperature.

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## I. INTRODUCTION

Service conditions for rubber products often demand satisfactory performance regardless of the temperature. For instance, tires must operate winter and summer, in the Arctic regions and in the Tropics. What is often more important, they must also withstand the elevated temperatures which are produced by the flexing of the tire itself. It is not unusual for tires to reach a temperature of from  $80^{\circ}$  to  $90^{\circ}$  C. on long runs at high speeds. On the other hand, there are conditions where rubber parts are subjected to extremely low temperatures, for instance, rubber used for aeronautical instruments, pilot balloons, and for shock absorber cord and tubing on airplanes. Accordingly the properties of rubber compounds at different temperatures are of vast practical importance in addition to their theoretical interest.

It is a matter of common knowledge that rubber compounds are affected by temperature and that the tensile properties at one temperature differ from those at another. Considerable data are available on the effect of temperature over a range such as is encountered in

ordinary testing,<sup>1</sup> but for temperatures above and below this range, very few results have been published. Probably the most complete results given for the effect of high temperatures are those published by Van Rossem.<sup>2</sup> These data, however, cover rubber-sulphur mixes only.

Breuil<sup>3</sup> gives some data for a range of temperatures from  $-10^{\circ}$  to  $+100^{\circ}$  C.

Le Blanc and Kroege<sup>4</sup> give considerable data on the effect of low temperatures. Their results also deal largely with rubber-sulphur mixes, although they point out that the presence of organic accelerators did not change their results appreciably.

It was the purpose of this investigation to cover a larger field than has been covered by previous investigators, both in regard to the types of compounds used and the temperatures employed. With this aim in view, the tensile properties of five typical rubber compounds, in addition to a rubber-sulphur mixture, were tested at temperatures from  $-70^{\circ}$  to  $+147^{\circ}$  C.

## II. RUBBER COMPOUNDS USED

The six compounds used in this work were chosen so that each was distinctive in some respect. Their composition and conditions of cure are given in Table 1.

TABLE 1

Ingredients	Compound letter					
	A	B	C	D	E	F
	Parts by weight					
Rubber.....	92.5	100.0	100.0	100.0	100.0	100.0
Sulphur.....	7.5	1.91	3.0	3.0	3.0	6.53
Zinc oxide (Kadox).....		1.03	130.0	5.0	5.0	18.75
Carbon black (Micronex).....				40.0		34.85
Mercaptobenzothiazole.....			.6	.6	.6	
Tetramethylthiuramdisulphide.....		.26				
Hexamethylene tetratramine.....						1.00
Stearic acid.....			3.0	3.0	3.0	
Mineral rubber.....						10.9
Palm oil.....						6.53
Reclaimed rubber (whole tire).....						38.1
Whiting.....					60.0	
Cures.....	90 minutes at $147^{\circ}$ C. ( $296.6^{\circ}$ F.)	25 minutes at $125^{\circ}$ C. ( $257^{\circ}$ F.)	45 minutes at $141^{\circ}$ C. ( $286^{\circ}$ F.)	45 minutes at $141^{\circ}$ C. ( $286^{\circ}$ F.)	30 minutes at $141^{\circ}$ C. ( $286^{\circ}$ F.)	50 minutes at $145^{\circ}$ C. ( $293^{\circ}$ F.)
Rubber by volume—per cent..	96.5	99.0	79	79	79	* 69

\* Includes 60 per cent rubber content of the reclaim.

<sup>1</sup> Dinsmore, Ind. and Eng. Chem., **17**, p. 540; 1925. Bruni, India Rubber World, **63**, p. 497; 1923. Nuckolls, Proc. A. S. T. M., **22**, p. 541; 1922. B. S. Circular No. 38. Rupert, India Rubber World, **76**, p. 139; 1927; and unpublished reports.

<sup>2</sup> J. Soc. of Chem. Ind., **45**, p. 68T; 1926.

<sup>3</sup> Breuil, Le Caoutchouc et la Gutta Percha, **7**, p. 4073; 1910.

<sup>4</sup> Kolloid Zeitschrift, **37**, p. 205; 1925.



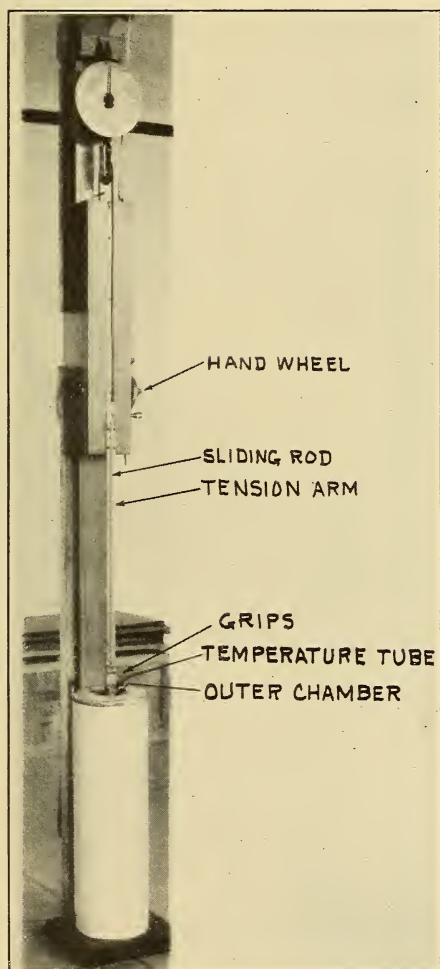
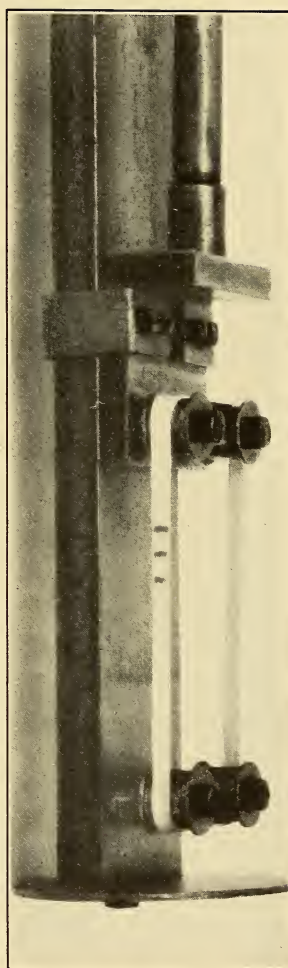


FIG. 1

*Apparatus used in testing rubber compounds at high and low temperatures. Machine shown in raised position*



*Enlarged view of four-spool grips with ring sample in position*

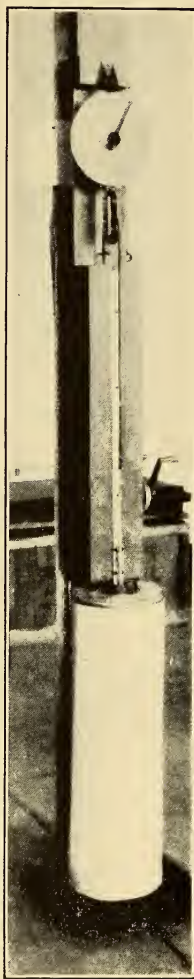


FIG. 2.—Apparatus used in testing rubber compounds at high and low temperatures. Tension arm shown lowered into temperature chamber

The first, or *A*, compound was chosen because of its theoretical interest and because it has been used by other investigators.

The *B*, or high rubber, compound contains approximately the maximum amount of rubber which is practical in a press cured compound. It also contains an organic accelerator and a low percentage of sulphur which distinguish it from *A*.

The main feature of the *C* compound is the high percentage of zinc oxide.

The *D*, or high carbon black, compound is similar to tread compounds.

In the *E* compound, whiting is the distinctive ingredient.

The *F* compound contains both zinc oxide and carbon black and, in addition, quite a large percentage of reclaimed rubber.

The compounds were cured in sheets averaging about 0.015 inch in thickness, by molding between aluminum plates.

The time of cure (90 minutes) for the *A*, or rubber-sulphur, compound is the same as for the intermediate compound used by Van Rossen. In this work it proved to be slightly below an optimum cure. The other compounds were cured to obtain maximum tensile strength at  $+23^{\circ}\text{C}$ .

### III. METHODS AND APPARATUS

Ring-shaped specimens, 4 inches in circumference (inside) and 0.1 inch wide, were used in the tests. The apparatus employed (figs. 1 and 2) operates as follows: With the machine raised as shown in Figure 1, the ring-shaped specimens are placed over the four spool grips at the bottom of the tension arm. Then the machine is lowered, so that the tension arm enters the inner chamber or tube. The desired temperature is maintained by liquids in the insulated outer chamber, which are heated or cooled as required.

The specimens were stretched and stress-strain data obtained by turning the handwheel which raises the sliding rod, and reading the balance at elongations shown on the vertical scale. A testing speed of approximately 20 inches per minute was maintained by timing with a stop watch as the specimen was stretched.

For temperatures below  $-10^{\circ}\text{C}$ ., the cooling medium was carbon dioxide snow and acetone; between  $-10^{\circ}\text{C}$ . and room temperature, salt and ice. For higher temperatures, oil heated by an electrical resistance wire was employed.

Temperatures in the inner tube were measured with a thermocouple at three locations—at the bottom, near the middle, and about 6 inches from the top of the tube. This was done before each specimen was tested and the deviation from the desired temperature was in all cases within  $\pm 1^{\circ}\text{C}$ . As a check on these readings temperatures were measured from time to time with a calibrated thermometer. In



order to maintain the uniformity of temperature throughout the length of the tube, it was necessary to stir the heating or cooling bath continuously.

In making these tests the sample was kept at each testing temperature long enough for it to become uniformly heated or cooled to that temperature. The time required was determined by the use of a thermocouple cemented between two test specimens placed in the inner tube. At the low temperatures, which required the longest period to reach the testing temperature, 5 minutes was found to be sufficient and for uniformity approximately this period was used throughout. To reduce oxidation at the high temperatures the specimens were given a preliminary heating for  $4\frac{1}{2}$  minutes under mercury and then transferred to the grips and lowered into the test chamber for  $1\frac{1}{2}$  minutes before testing. Approximately 10 seconds were required to raise the machine and transfer the specimens from the preliminary bath to the grips, after which a minute was required for all parts to again reach the testing temperature.

In order to avoid difficulties which were encountered at low temperatures due to moisture freezing on the spool grips and other parts of the machine, check tests were made at  $-30^{\circ}$  C. and lower temperatures with alcohol in the test chamber. This eliminated the formation of ice on the machine parts and gave somewhat more consistent results. As far as could be determined, the presence of alcohol had little effect on the physical properties of the rubber. The effect, if any, was to stiffen the compounds slightly. In the alcohol bath the time required for samples to reach the testing temperature was less than in air and 2-minute exposure periods instead of the 5-minute periods were found to be sufficient.

Stress-strain data based on the dimensions of each individual specimen were obtained on each of the rubber stocks at different temperatures as follows:  $+147^{\circ}$ ,  $+130^{\circ}$ ,  $+100^{\circ}$ ,  $+70^{\circ}$ ,  $+40^{\circ}$ ,  $+23^{\circ}$ ,  $+10^{\circ}$ ,  $-10^{\circ}$ ,  $-30^{\circ}$ ,  $-50^{\circ}$ ,  $-60^{\circ}$ , and  $-70^{\circ}$  C. At each temperature from 3 to 8 test specimens were broken and the results averaged.

#### IV. RESULTS OF TESTS

The results of these tests are shown graphically in three ways. Figures 3 to 8 show the effect of temperature on the tensile strengths and elongations at break for the six compounds. Figures 9 to 14 show stress-strain data for the six compounds. Each line on these charts (figs. 9 to 14) traces the variations in stress with temperature for a particular elongation:

In the first set of curves (figs. 3 to 8) it will be noted that at  $-70^{\circ}$  the elongations are zero for all compounds and that the highest per cent elongations in all cases is reached at a temperature between about  $100^{\circ}$  and  $120^{\circ}$  C. At higher temperatures there are decided



decreases in the elongation. There is a very rapid rise in the elongations between  $-70^{\circ}$  (at which point the rubber was rigid) and  $-55^{\circ}$  C. The data between these two temperatures are uncertain,

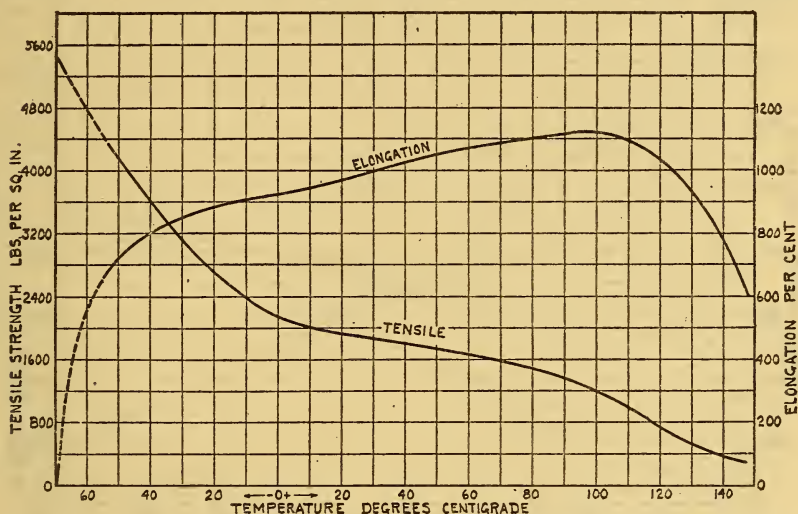


FIG 3.—Compound A (rubber sulphur). The effect of temperature on the tensile strengths and elongations at break

and the curves are shown as broken lines. The temperature at which all stocks seemed to lose their elasticity was about  $-60^{\circ}$  C.

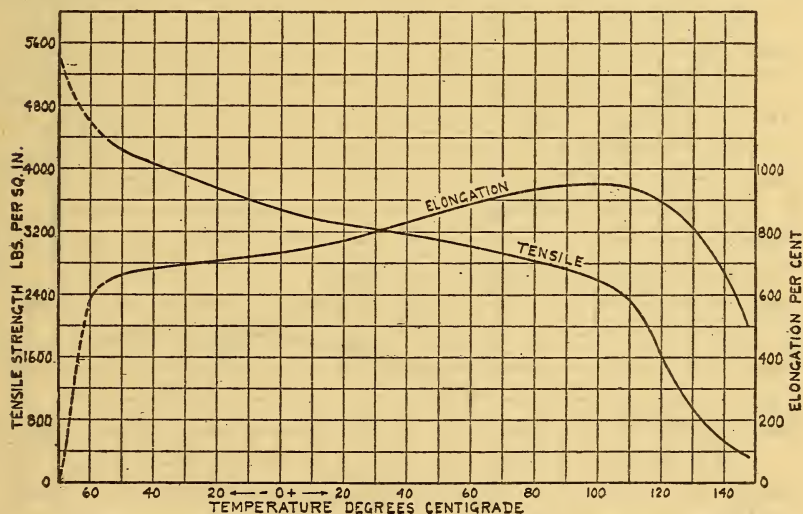


FIG. 4.—Compound B (high rubber). The effect of temperature on the tensile strengths and elongations at break

It appears that the tensile strengths of compounds high in rubber, such as A and B (figs. 3 and 4), are less affected by temperature changes in the middle of the range than are those which are compounded.

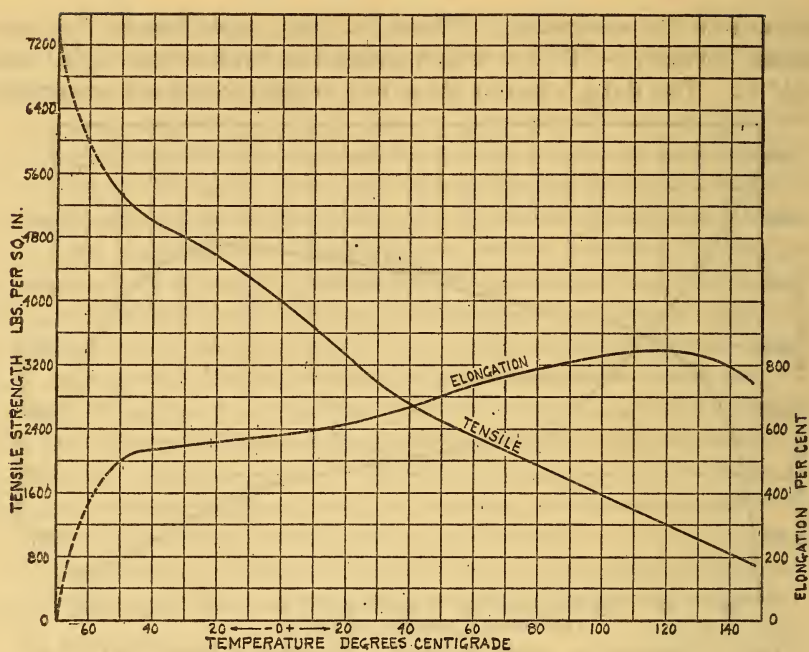


FIG. 5.—Compound C (zinc oxide). The effect of temperature on the tensile strengths and elongations at break

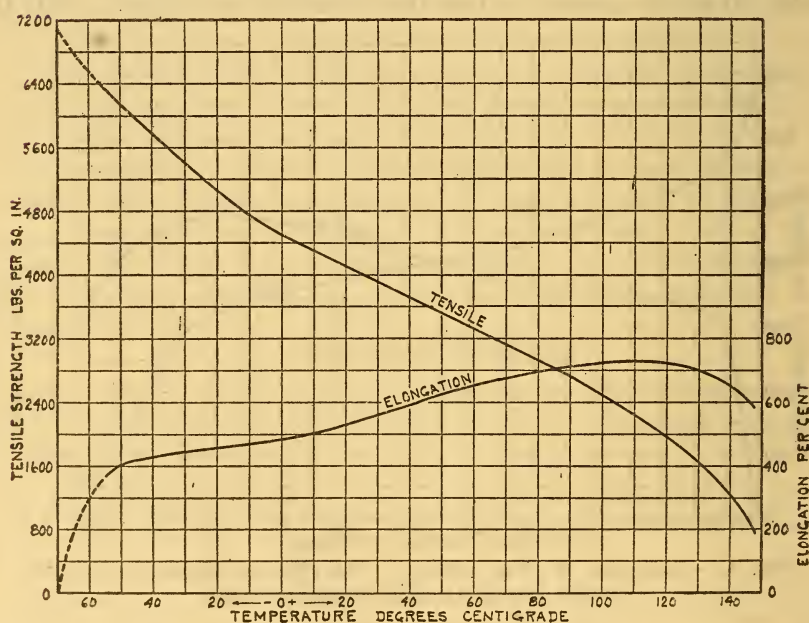


FIG. 6.—Compound D (carbon black). The effect of temperature on the tensile strengths and elongations at break

The second set of curves (figs. 9 to 14) indicate that the stiffening of the compounds as the temperature is lowered is very marked,

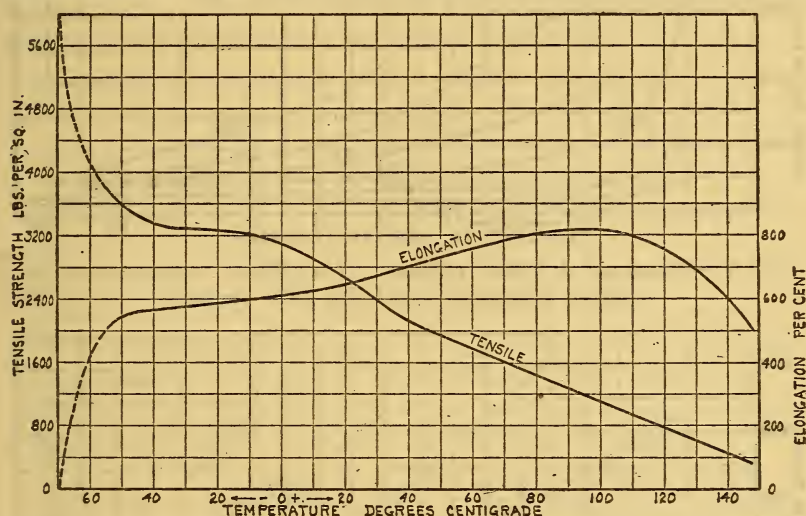


FIG. 7.—Compound E (whiting). The effect of temperature on the tensile strengths and elongations at break

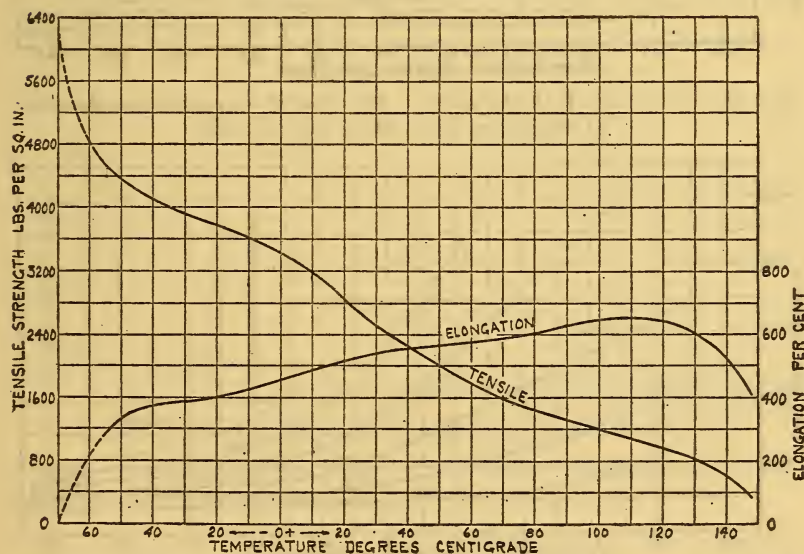


FIG. 8.—Compound F (reclaimed rubber). The effect of temperature on the tensile strengths and elongations at break

particularly at the higher elongations. For any given stretch the rate at which the stress changes with the temperature increases as the temperature decreases. These curves also indicate that over the



middle of the temperature range high-rubber compounds (A and B) are less affected by differences in temperature than are compounded stocks.

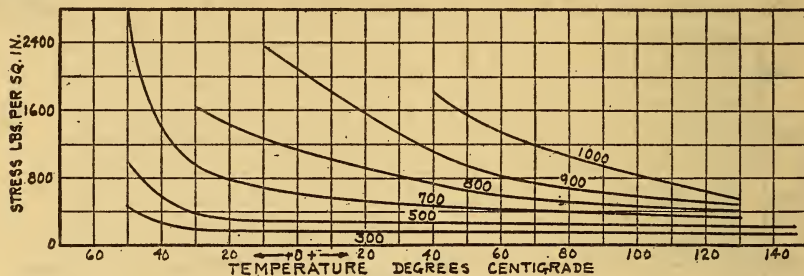


FIG. 9.—Compound A (rubber sulphur). The effect of temperature on the stress at elongations from 300 to 1,000 per cent

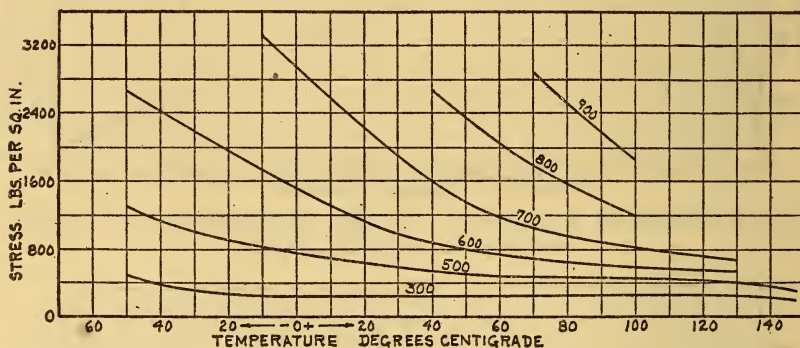


FIG. 10.—Compound B (high rubber). The effect of temperature on the stress at elongations from 300 to 900 per cent

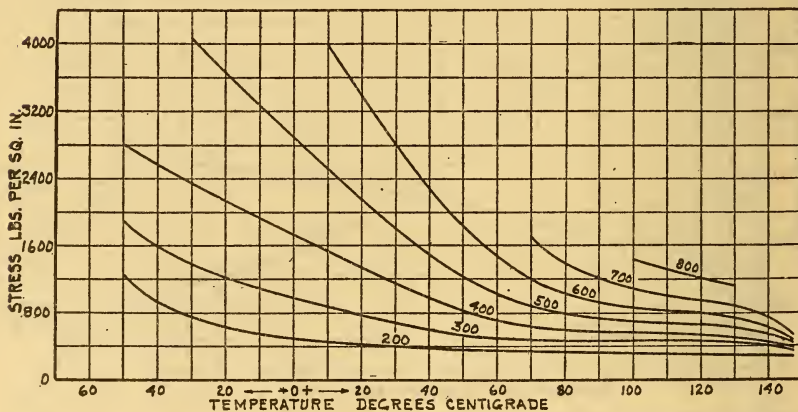


FIG. 11.—Compound C (zinc oxide). The effect of temperature on the stress at elongations from 200 to 800 per cent

In Figure 15 the data showing tensile strengths and elongations at break (figs. 3 to 8) have been combined to show the influence of temperature on the tensile product; that is, the product of the



tensile strength in pounds per square inch and the per cent elongation. The significant feature of these curves is the very nearly constant tensile product of the high rubber, or *B*, compound below about 80° C. This is true to a lesser extent of the carbon black compound *D*. The tensile product of the rubber-sulphur compound *A* is the most affected over the temperature range.

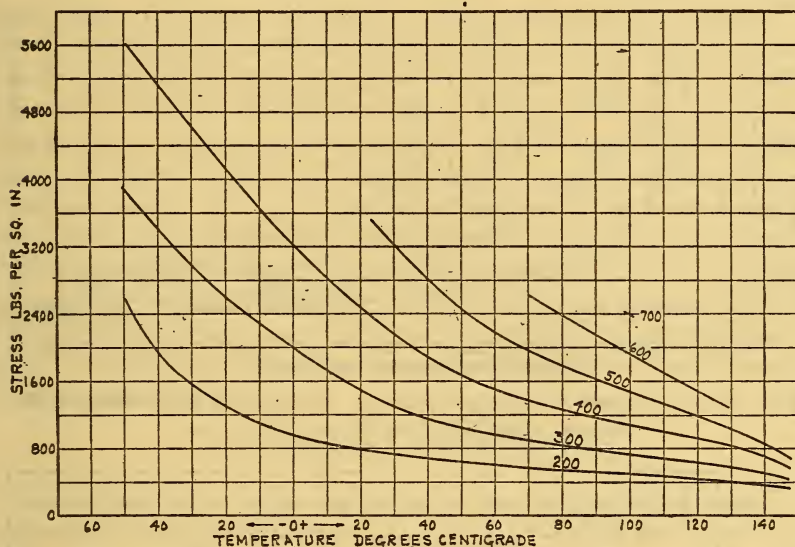


FIG. 12.—Compound *D* (carbon black). The effect of temperature on the stress at elongations from 200 to 700 per cent

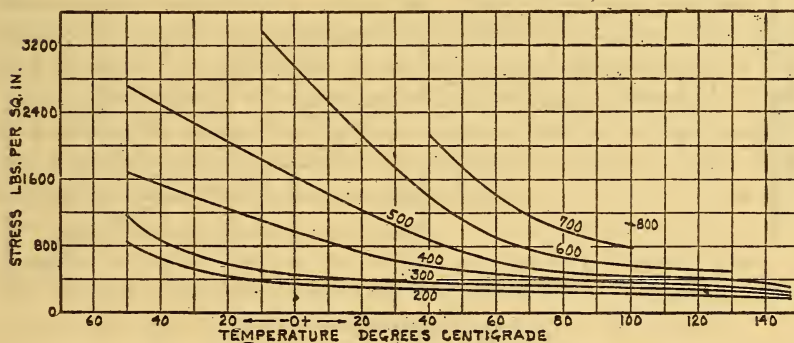


FIG. 13.—Compound *E* (whiting). The effect of temperature on the stress at elongations from 200 to 800 per cent

As a basis of comparison, some conclusions are drawn as to the changes in tensile strength and elongation over that part of the temperature range where the curves are all comparatively straight, namely, from -20° to +100° C. The least change in tensile strength between these temperatures is found in the high rubber compound *B* in which the tensile strength is 30 per cent less at +100° C.

than at  $-20^{\circ}\text{C}$ . The other compounds show tensile strengths 50 to 70 per cent less at  $+100^{\circ}\text{C}$ . than at  $-20^{\circ}\text{C}$ . The elongations are from 25 to 60 per cent higher at  $+100^{\circ}\text{C}$ . than at  $-20^{\circ}\text{C}$ ., the rubber-sulphur compound *A* and the high rubber compound *B* showing the smallest changes. If it is assumed that there is a straight-line relation throughout this temperature range, the following

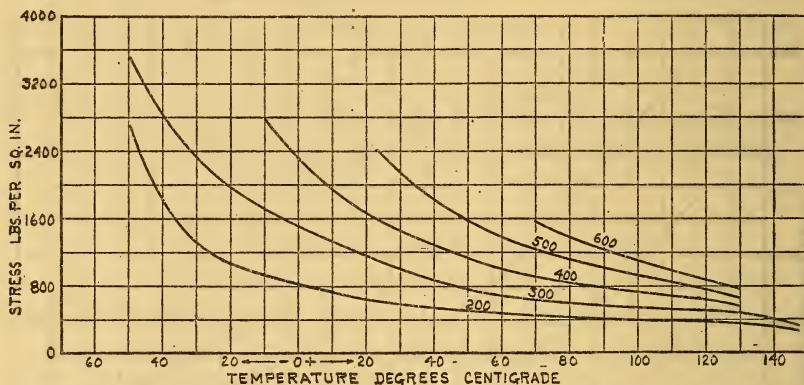


FIG. 14.—Compound *F* (reclaimed rubber). The effect of temperature on the stress at elongations from 200 to 600 per cent

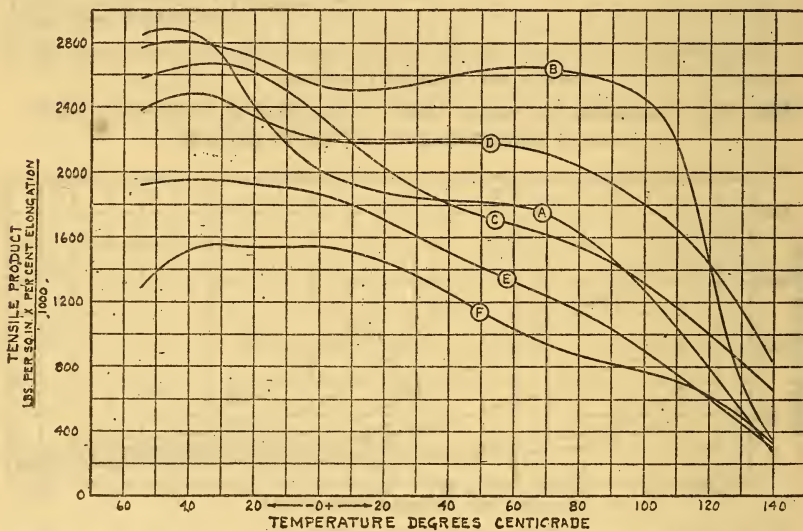


FIG. 15.—Effect of temperature on the tensile products

differences would be observed under ordinary testing conditions: Considering a minimum temperature of  $+18^{\circ}\text{C}$ . ( $64.4^{\circ}\text{F}$ .) and a maximum of  $+32^{\circ}\text{C}$ . ( $89.6^{\circ}\text{F}$ .), tensile strengths would be expected from 3.5 to 8 per cent less at  $32^{\circ}\text{C}$ . than at  $18^{\circ}\text{C}$ . and elongations from 3 to 7 per cent greater. Although it was not the purpose of this investigation to obtain precise data over this temperature

range (18 to 32° C.) the figures are in general agreement with those obtained in investigations which covered the range more completely.<sup>5</sup>

In making these tests several incidental features concerning the properties of rubber at low temperatures were noted. A stress-strain curve representing the properties of rubber just below -60° C. has a peculiar shape. The tensile stress rises very rapidly to a maximum with very little elongation. The heat generated during this period seems sufficient to soften the rubber, and on further stretching the stress drops and then rises to another peak at the breaking point. At -75° C. the rubber compounds were sufficiently rigid that they could be broken by a sharp blow. Some of the specimens were stretched several hundred per cent before being lowered into the temperature tube at -70°. They became rigid and, on a further application of load, broke with a very slight elongation. The tensile strengths of the stretched specimens were lower than those of the unstretched specimens. Cooling samples of rubber until they became rigid and then allowing them to come to room temperature apparently had no effect on the physical properties even after the cycle had been repeated as many as 12 times.

## V. GENERAL CONCLUSIONS

The results show that temperature has a very pronounced influence on the tensile properties of rubber. At low temperatures rubber becomes rigid with an increased tensile strength, and at high temperatures it possesses practically no tensile strength. The various rubber compounds investigated show the same type of behavior with changes in temperature. Differences due to compounding ingredients are differences in degree rather than in kind.

The magnitude of the changes in rubber compounds due to temperature shows that the effect of temperature is a feature which should be given consideration in the design of rubber articles which are used at other than normal temperature.

WASHINGTON, September 20, 1927.

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<sup>5</sup> B. S. Circular No. 38, Dinsmore, Ind. and Eng. Chem., 17, p. 540; 1925.

